

**Appendix II**

**Preliminary Design Criteria for  
CWS Filter Processing**



ATTACHMENT A

PRELIMINARY DESIGN CRITERIA  
FOR  
CWS FILTER PROCESSING

by  
U. M. Anderson

MP-25-66

July 15, 1966

THE DOW CHEMICAL COMPANY  
Rocky Plats Division  
Golden, Colorado

U. S. Atomic Energy Commission Contract AT(29-1)-1106

Distribution:

H. G. Moss

cc:

K. V. Best  
J. G. Epp  
D. C. Farrell  
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REVIEWED FOR

JUL 18 1972

PATENTABLE  
MATERIAL

*None found  
Official*

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## 1.0 INTRODUCTION

Currently there is no process or equipment on hand to process the backlog or current generated CWS filters from Buildings 71 and 76. This report proposes a process and the necessary equipment to work off this backlog.

## 2.0 SUMMARY

The proposed process for recovering plutonium from CWS filters consists of the following:

- A. Remove frame from filter media using hand tools and tear filter media apart.
- B. Place filter media in a geometrically unfavorable vessel containing 13 M  $\text{HNO}_3$ ,  $\text{CaF}_2$  and  $\text{CeH}_2\text{O}_3$  at 90° C.
- C. Leach material from 1 to 4 hours using mechanical agitation.
- D. Dump material out of vessel onto a screen backed by a roughing filter.
- E. Spray wash the remaining filter media with .35 N  $\text{HNO}_3$  while on the aforementioned screen.
- F. Pull solid as dry as possible, with vacuum source on filter.
- G. Barrel solids with magnesia cement for burial.
- H. Transfer liquid to storage tank for further processing by ion exchange.
- I. (1) Bag out metal frames for burial.  
(2) Bag out wooden frames for incineration.

The proposed CWS filter processing system consists of the following: (Figure 1)

- A. Drybox, air lock, bag and drum take out, etc.
- B. Disassembly area
- C. Steam jacketed, agitated kettle, with removable top.
- D. Fume removal system.
- E. Screens, and filters
- F. Spraying system
- G. Hopper
- H. Holding tanks
- I. Batch make-up tanks

### 3.0 GENERAL

#### 3.1 Description

Flanders & Cambridge C.W.S. Type F Filter.

Size: 24" x 24" x 11-1/2"

Weight: ~ 6 lbs.

Filter media: glass-asbestos

Separator: aluminum or asbestos

Frame: wood or cadmium plated steel

#### 3.2 Generation and Backlog

As of June, 1966, 463 filters containing an estimated 34.6 Kg are on hand awaiting processing. Current generation is approximately 20 filters per month (300-1000 gm Pu.)

### 3 Processing

#### 3.3.1 Leaching

To promote dissolution of the Pu contained on CWS filters it is necessary to both leach and agitate. While leaching some filters present a foaming problem, therefore the leaching vessel must have ample capacity to handle this situation.

Those filters coming from the 71 Building incinerator off-gas system are of the aluminum separator type. This aluminum when dissolved in  $\text{HNO}_3$  liberates large quantities of  $\text{NO}$ ,  $\text{HF}$ , and  $\text{H}_2$  fumes. The system must therefore be capable of handling said vapors.

#### 3.3.2 Filtering

The filter media in the leach solution do not settle out sufficiently to allow decantation of liquid. Therefore it is necessary to filter the slurry on a large enough surface area that reasonable flow rates can be maintained. Accumulation of cake slows filtering down considerably on this material.

#### 3.3.3 Drying

Excess liquid must be removed from the material to prevent leakage during shipping. Some leakage is however tolerable if the barrel is properly packaged with magnesia cement.

Therefore the solid will be pulled dry by vacuum and packaged directly with magnesia cement.

#### 4.0 DESIGN BASIS

The processing facility should be capable of leaching 1 filter per 8-hour period. Processing vessel should have capacity to leach a minimum of 1/2 CWS filters per cycle, with ample over-capacity for foaming. Assuming 30 to 35 gallons of liquid are needed per filter, a 20 to 25 gallon vessel should be acceptable. The vessel must have heat capacity to raise the temperature of the acid from 23° C to 100° C in less than 30 minutes and instrumentation to control the temperature in the 50° C to 100° C range.

The filter system should be of sufficient capacity to hold contents of leaching vessel and so designed to filter contents in less than one hour.

Makeup tanks and storage tanks must be of sufficient size to provide uninterrupted service on a three-shift basis.

#### 5.0 EQUIPMENT

##### 5.1 Drybox

A drybox is required to contain the system. An airlock is required to introduce a whole filter including outer package into the line. A smaller airlock is needed to put supplies into the line. Bag and drum take outs are required to remove filter frames and filter media.

### 5.2 Disassembly Area

A table is necessary to place filter on while removing the filter media from the frame. Electric hand tools will be used for this task. The filter media will be torn apart and placed in the kettle from this point.

### 5.3 Leaching Vessel

The leaching vessel should be an open top, steam jacketed, baffled, tilting kettle. It must be capable of heating 13 M  $\text{HNO}_3$  from  $20^\circ \text{C}$  to  $100^\circ \text{C}$  in less than 30 minutes. A removable top complete with agitator and fume removal connection are necessary. The kettle should have a capacity of approximately 25 gallons.

### 5.4 Solid-Liquid Separation

The filtering system should be so constructed that a screen basket can be placed over the filter to allow for easy removal of the solid material. The filter should have storage capacity to hold the bulk of the slurry.

### 5.5 Fume Removal

The system should include a wet scrubber to remove nitrous fumes,  $\text{HF}$ ,  $\text{H}_2$ , and  $\text{SiF}_4$  from the reactant mixture.

## **Appendix JJ**

### **Analysis of Waste Drums No. 771-7959 and 771-7961 Returned from the National Reactor Test Site**



Reports on Drums Returned  
to Rocky Flats from INEEL



# THE DOW CHEMICAL COMPANY

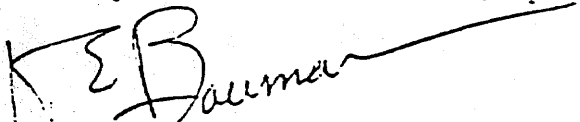
ROCKY FLATS DIVISION  
P. O. BOX 888  
GOLDEN, COLORADO 80401

December 16, 1971

Mr. F. E. Abbott  
Manager, RFAO, USAEC

ANALYSIS OF WASTE DRUMS NO. 771-7959 AND 771-7961,  
RETURNED FROM THE NATIONAL REACTOR TEST SITE.

Transmitted herewith are two copies of the report, "Analysis  
Of Rocky Flats Waste Barrels," prepared by A. K. Williams.

  
H. E. Bowman  
Assistant General Manager  
for Operations

HEB:mmm  
Orig. and 1cc - Mr. Abbott  
Enc.

cc:

R. E. Hayne - Dow, Rocky Flats  
M. E. Hughes - Dow, Rocky Flats  
J. B. Owen - Dow, Rocky Flats  
✓ M. A. Thompson - Dow, Rocky Flats  
J. F. Willging - Dow, Rocky Flats  
A. K. Williams - Dow, Rocky Flats

# ANALYSIS OF ROCKY FLATS WASTE BARRELS

A. K. Williams

December 14, 1971

Distribution:

F. E. Abbott	- AEC, RFAO
H. E. Bowman	- Dow, Rocky Flats
R. E. Hayne	- Dow, Rocky Flats
M. E. Hughes	- Dow, Rocky Flats
J. B. Owen	- Dow, Rocky Flats
M. A. Thompson	- Dow, Rocky Flats
J. F. Willging	- Dow, Rocky Flats
A. K. Williams	- Dow, Rocky Flats

# ABSTRACT

This report covers the activity at both Rocky Flats and the National Reactor Testing Station (NRTS) in returning drums numbered 771-7959 and 771-7961 to Rocky Flats. The total quantity of plutonium found in these two drums was 1589 grams. Comparisons are made between various counting systems used to assay the drums and the final Rocky Flats' analysis.

## INTRODUCTION

In January 1971, two Rocky Flats waste drums, Nos. 771-7959 and 771-7961, were shipped to the Idaho Nuclear Corporation (now Aerojet Nuclear Corporation [ANC]) for storage. At that time, it was indicated that each drum contained zero grams plutonium. A subsequent review of Rocky Flats (RF) drum counting data (March 1971) indicated that these barrels could contain appreciable quantities of plutonium. The two drums were located by ANC and isolated. Gamma measurements were made by J. E. Cline of ANC, and he reported that drum 771-7959 contained a minimum of 600 grams plutonium and a maximum of 1700 grams plutonium; and, drum 771-7961 a minimum of 370 grams plutonium and a maximum of 1100 grams plutonium. This information was received at the Rocky Flats Area Office on June 7, 1971 in a memo from W. L. Ginkel (IDO) to F. E. Abbott (RFAO). A team consisting of Dow, RFAO, and ALO personnel then visited IDO on June 30, through July 1, 1971 to put together a plan to repackage the drums to allow their return to RF using existing transportation permits. This report summarizes the activities which transpired in repackaging the drums for return. It also presents comparisons in data between counting systems used at NRTS by Gulf-Atomic Mobile Assay System (GAMAS) and ANC, and RF. These are compared with analytical data from RF. The official values reported are the RF analytical results.

## CONCLUSIONS

In reviewing the contents of these drums and correlating dates and RF practices and procedures for loading and counting drums of waste and recoverable residues, the following conclusions were derived.

1. The plutonium scrap recovery area had been started up on a limited basis (dissolution had been allowed to start) when the substantial loss in plutonium was reported in the December 1970 inventory. The area was shut down and cleanup of the glovebox lines started. The major portion of materials in these drums were residues cut out from the dissolution lines, 2 and 3. ---
2. The sludge and filter media found in the drums should have been can counted and assigned a value before placing in a drum. There was a violation of procedures for those packages containing sludge and FulFlo<sup>®</sup> filters.
3. The drum counter produced count ratios which could not be handled by the computer program; thus, a negative number was printed.
4. The drum count sheets were not reviewed by a technical person as they were generated. The sheets showing zero grams plutonium allowed for shipping these drums to Idaho as waste.

5. Gamma survey results on these drums did not alert anyone to thinking that these drums contained large quantities of plutonium. This is probably due to some drums showing high gamma readings from americium.
6. Splitting of drums can be done safely at Idaho with experienced personnel following a predetermined plan.
7. Analysis by the GAMAS and ANC systems compare favorably with the final analysis at RF.

## DISCUSSION

### 1. Preliminary Analysis

Because of anomalies in drum count results involving negative numbers and inventory problems in the plutonium scrap recovery plant in December 1970 and January 1971, a search was made by RF to determine if there were any drums sent to Idaho which may have contained substantial quantities of plutonium. In reviewing 27,800 barrel count sheets (for both RF fire waste and regular production waste) generated in the period June 1969 through February 1971, five drums were identified as being suspicious. Three of these drums were generated a number of months earlier and were buried in trenches. Two drums were generated in January 1971 and these were located by ANC and isolated from the waste storage area. These drums were identified as combustibles by RF. The drum count sheets for these two drums are shown in Figures 1 and 2. In reviewing these sheets, we found at RF that the gamma to neutron ratio by calculation could lead to a print of negative numbers, and that when the data was fed to the computer, the plutonium content would be reported as zero. In the past, small negative numbers (such as -1.0) could be expected in some drums with low counts. These two drums had high counts and large negative numbers. It was also noted that the scaler used in the counting system could exceed its highest number, flip over, and start again at zero. We concluded that this

occurred when these two drums were counted. Recognizing the problem, corrective action was taken to prevent the scaler from exceeding its limits. The computer was reprogrammed so a plutonium value would not be printed if the count ratios were outside present limits. These corrective actions were completed in February 1971. In addition to the above, a qualified technical man now reviews each drum count sheet to ensure that any strange count ratios are detected immediately. Besides preventing additional accidental shipments to waste, this technical review will also minimize assigning improper plutonium values to residues awaiting processing in the RF scrap recovery plant.

The gamma survey on these drums showed 32 mr/hr for 771-7959 and 17 mr/hr for 771-7961. These values indicated that the waste was active and could contain measurable quantities of plutonium. We surmise that at the time, the gamma activity was thought to be from americium.

Analysis by J. E. Cline of ANC showed that drum 771-7959 contained from 600 to 1700 grams plutonium and that drum 771-7961 contained from 370 to 1100 grams plutonium. Radiographs indicated an undefined high density area in drum 771-7959 which appeared to contain a large quantity of plutonium. Based on these results, it was decided to split

the drums at NRTS so that they could be returned to RF. The contents of each drum appeared to be too high to allow return to RF under existing transportation permits. A team consisting of Dow, RFAO, and ALO personnel visited IDO on June 30 and July 1, 1971, to devise a plan with IDO and ANC personnel to split the drums and return them to RF.

2. Plan for Opening RF Drums 771-7959 and 771-7961

Personnel involved in the planning at the June meeting were as follows:

Dow-RF	A. K. Williams, M. A. Thompson, J. B. Owen
RFAO	T. C. Jones
ALO	W. B. Johnston
IDO	G. Wehman, T. W. Asbury, B. D. Johnson, P. G. Voilleque, M. Hankins, B. Estes, B. L. Schmalz
ANC	B. R. Baldwin, J. W. McCaslin, R. B. O'Brien, D. P. Halls, W. W. Hickman, L. D. Hanson, J. Hanny

After preliminary discussions regarding operational safety and possible methods and locations for opening the drums, a plan was developed by contractor and AEC personnel. This plan was approved by all parties concerned on July 1, 1971. The plan called for the work to be performed in the ARA-1 hot cell, since this cell had one stage of HEPA filtration.

Rocky Flats personnel would construct a plastic tent inside the cell which would contain an additional stage of HEPA filtration. The active packages which were thought to be small, would be placed in either a modified 6M container (DOT permit 5791) or a 1518 container, when the drum was split. The purpose of the splitting was to reduce the plutonium content of each drum to less than 200 grams since this was the limit of DOT permit 5948. The modified 6M container (DOT permit 5791) can contain up to 4.5 kg plutonium for compounds having an H:X ratio  $\leq 3$  for Class II shipment. Upon returning to RF, it was found that this permit could not be used, since the plutonium must not decompose at temperatures up to 750°F. Because of this, a special exemption was requested for the 2030 container (DOT permit 5332) for up to 1500 grams per container.

Splitting of the drums was scheduled for the week of July 19, 1971. Upon completing the plan and obtaining agreement from everyone concerned, RF personnel began making arrangements to collect and ship the necessary materials to do the drum splitting at NRTS. A copy of the detailed plan is on file at Rocky Flats. Copies of this plan are also available at ALO, IDO, and ANC.

### 3. Drum Splitting

The Rocky Flats' team arrived in Idaho on July 19, 1971. Previous to their arrival, all needed supplies had been shipped by RF and received by ANC. The Rocky Flats' team consisted of the following personnel:

A. K. Williams	- Chemical Operations
J. B. Owen	- Waste Management
M. E. Hughes	- Chemical Operations
R. E. Hayne	- Health Physics

Upon arrival at the facility (ARA), a safety review was conducted by L. D. Hanson. A health physics indoctrination was then held at Central Facilities. Upon completion of the indoctrination, a health physics survey of alpha and gamma activity in ARA-1 was conducted by RF. The survey results were then compared with previous surveys taken by ANC health physics personnel. After comparing and agreeing on the activity levels, RF personnel then constructed a plastic house inside the cell. Four filters (2 feet by 2 feet by 1 foot) were needed to provide adequate airflow from the house. The house was completed the evening of July 19, and plans were made to open and repack the drums on July 20.

On July 20, the procedures to be used were again reviewed by RF personnel with L. D. Hanson at ANC. At that time, it was agreed that a dry run was not required. The house in ARA-1 was constructed so that the entire operation could be observed from the operating side. Besides those from ANC, observers from AEC-Washington - G. L. Daly, ALO - W. Holmes, and IDO - G. Wehman, observed all operations. An ANC health physics technician provided backup support, and L. D. Hanson remained in charge of all emergency equipment.

All Dow personnel were dressed to prevent skin contamination and wore full face mask respiratory protection during the drum splitting and cleanup operations.

After agreement was reached between ANC and Dow personnel on the adequacy of the procedures to be used, the first drum was transferred to Dow and placed inside the plastic house.

J. B. Owen and M. E. Hughes performed the drum splitting operation, R. E. Hayne performed the necessary health physics services, and A. K. Williams coordinated the effort and mapped the drums.

In the first drum (771-7959), the following observations were made.

- a. Upon opening the lid, alpha contamination was found on the inside surfaces of the drum. Because of this, all packages, as they were removed, were resealed in an additional bag. In some instances, packages were double bagged.
- b. The top 12 inches of the drum was empty.
- c. The top package consisted of tygon tubing which read 20 mr/hr and occupied the area to approximately a 6 inch depth.
- d. The next third of the drum contained nine packages which varied from 4 mr/hr to 250 mr/hr in gamma activity. Two of the packages were found which indicated high gamma levels. One package showed 250 mr/hr and appeared to consist of wet wiping papers. Some free liquid was observed in the bag. This bag was found at the center of the drum, directly under the top package of tygon tubing. The ticket on the package indicated that its contents came from Line 2, which is a dissolution line at RF. The second package was located 10 inches from the side of the drum about halfway down. This package showed 190 mr/hr gamma and contained about 1/2 inch of sludge in a quart plastic carton, and wiping papers.

- e. The bottom third of the drum contained packages which varied from 5 mr/hr to 22 mr/hr gamma.
- f. Material was identified as coming from Lines 2, 3, 5, and 21.

Based on these observations, each of the two high level gamma packages was placed in a 2030 container. The first was placed in container No. 771-7959B and the second package in container No. 771-7959A. The containers were sealed, surveyed, and removed from the cell. Outside, they were again surveyed and transferred back to ANC. The containers were then sent to the GAMAS trailer for analyses. The remaining packages were placed in a new drum and inspected and sealed following the waste management procedures used at RF. The empty contaminated drum was enclosed in plastic bags, sealed, and placed in a RF crate for disposal.

After removing all material from the first drum from the building, the second drum, 771-7961, was brought in and transferred to Dow personnel. Upon opening this drum, the following observations were made.

- a. No contamination was found when the drum was opened.

- b. Two active packages were found. One was found on the side about halfway down and reading 125 mr/hr gamma. The second package was on the opposite side and 6-10 inches lower, and read 110 mr/hr gamma. No free liquids were visible in these two packages and the material appeared to be plastic and paper. No wet items were found in this drum. Each of the two active packages were placed in a 2030 container. The 125 mr package was put in container 771-7961A, and the 110 mr package was put in container 771-7961B.
- c. No contamination was found inside this drum. The contents were split by removing half of the remaining packages and placing them in a new drum (771-7961A). The original drum was then numbered 771-7961B.

These drums were then packed and inspected using RF waste management procedures. Each of the 2030 containers and drums were monitored and removed from the cell. The containers and drums were then resurveyed and transferred back to ANC. Each of the above were then sent to the GAMAS for analysis.

#### 4. Cleanup Operations

Upon completion of splitting operations, the plastic house was dismantled, and all items which were contaminated or

suspected of being contaminated, were placed in the RF crate. The crate was then sealed and prepared for disposal at the Idaho waste storage area. The remaining items were packaged for return to RF. After cleanup was complete, a health physics survey was taken of the ARA-1 cell and surrounding area. The survey results were compared with the initial survey and it was agreed between ANC and Dow that the operation did not further contaminate any area.

Upon completing the cleanup, Dow personnel returned to RF. Preliminary analysis by the GAMAS indicated that the drums each contained less than 200 grams and that each of the 2030 containers contained less than 1500 grams. The containers were then put in storage awaiting DOT approval for shipment to RF.

##### 5. Shipment to Rocky Flats

Approval to ship these containers to RF was received on July 27. Rocky Flats personnel then made arrangements to pack the containers into the approved outer container and seal them for shipment to RF. In the meantime, ANC had informed RF that three of the containers showed a pressure on the gages. (The inner container of the 2030 shipping package is essentially a pressure cooker equipped with a pressure gage.) Container 771-7959B showed a pressure of

7 psig, 771-7961A showed 7 psig, and 771-7961B showed 1 psig. Preparations were then made to vent the containers before preparing for shipment to RF. Since observations were not made on gage pressure at the time the containers were closed, there was some uncertainty on the meaning of the observed values. With these particular containers, problems are occasionally experienced with faulty pressure gages. Dow personnel vented the containers on August 13. The only one showing pressure relief was 771-7959B. Container 771-7961A remained the same, and 771-7961B was not vented. Health physics surveys during the venting operation showed no release of alpha contamination. The four containers were received at Rocky Flats on August 18, 1971. Each of the containers was analyzed in the RF drum counter to provide an estimate of the plutonium values and then given to the R&D Chemical Processing group for processing and further analysis. Details of this work is found in CRDL-950345-15-1.<sup>1</sup> A summary of the data in this report is as follows:

Container No. 771-7959A

The contents were sludge and rags. These were separated, analyzed by can counter and then

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<sup>1</sup>R. E. Giebel, R. G. Leebl, and J. A. Battaglino, "Plutonium Measurement in Idaho Waste Return," CRDL-950345-15-1, dated November 12, 1971 (internal report).

calcined and analyzed by X-ray. The analyses for this container were as follows:

GAMAS	- 270 grams
RF Can Counter	- 262 grams
RF X-ray on Ash	- 264 grams
RF Drum Counter	- 167 grams (estimate)

Container No. 771-7959B

The inner container showed 1 psig. Because of the history of pressure buildup at Idaho, the entire container was placed in the glovebox line. When the container was opened, no evidence of pressurization was found and the sealed plastic bags inside the container were not pressurized. The contents were found to be three filter pads containing plutonium bearing sludge, a FulFlo filter, a pint polyethylene bottle half full of wet sludge, and a second pint bottle 1/4 full of wet sludge, plastic and paper. The material was identified as coming from Line 2, and was packaged on January 18, 1971. A total of 70 ml of liquid was found which assayed 0.21 g/l Pu,  $5.3 \times 10^{-3}$  g/l Am, and  $2.2 \text{ N H}^+$ . The contents of the container were split into five separate packages and can counted. Each package was then calcined

and the ash sampled for X-ray analysis. The analyses of this container were as follows:

GAMAS	- 600-800 grams
RF Can Counter	- 715 grams
RF X-ray on Ash	- 974 grams
RF Drum Counter	- 628 grams (estimate)

Container No. 771-7961A

Although the gage on this container showed greater than 15 psig, attempts to vent the container resulted in no apparent pressure relief. The contents were found to be plastic, a towel, paper, sludge, plastic tubing, and some scrap metal. The analyses for this container were as follows:

GAMAS	- 243 grams
RF Can Counter	- 243 grams
RF X-ray	- 252 grams
RF Drum Counter	- 225 grams (estimate)

Container No. 7961B

The pressure gage was under 7 psig. The plastic bag inside the container was pressurized and contained filter sludge. Analyses for this container were as follows:

GAMAS	-	98 grams
RF Can Counter	-	106 grams
RF X-ray	-	99 grams
RF Drum Counter	-	85 grams (estimate)

The RF drum counter results on the 2030 containers are low in comparison to the others reported. These results must be considered estimates, since the containers did not approximate the standards and the plutonium was highly localized.

Although we originally intended to leave the three drums of remaining material at Idaho, these were returned to RF on October 18. This was done at the request of Roy Crouch, ALO, and agreed to by A. K. Williams, Dow. Analyses of these drums were as follows:

<u>Drum No.</u>	<u>GAMAS</u>	<u>RF</u>
771-7959	155	163
771-7961A	217	165
771-7961B	50	61

Comparisons of RF, GAMAS, and ANC analyses for the total drum contents are found in Table I.

TABLE I

Comparison of Analyses Between RF, GAMAS, and ANC

<u>Barrel No.</u>	<u>g Pu GAMAS</u>	<u>g Pu ANC</u>	<u>g Pu RF</u>
771-7959	1060 ± 180	600-1700	--
771-7961	560 ± 90	370-1100	--
771-7959 Divided Totals	1125 ± 120	---	1401
771-7961 Divided Totals	608 ± 60	---	577

#### CORRECTIVE ACTION

Many of the corrective actions were implemented within a month of these two drums being shipped. These were instituted because of problems involving drum splitting at RF which, in turn, led to searching for anomalies in drum counting as outlined previously.

1. A new high level drum counter has been installed and is currently in operation. This counter contains many improvements which should eliminate the chances for shipping additional drums containing significant quantities of plutonium.
2. The original drum counter now has mechanical stops to prevent the registers on the scaler from flipping over. This counter is now used for non-line generated waste and as a backup system for the new counter.
3. The computer program has been changed so that count ratios outside the prescribed limit cannot be calculated. The print-out on the count sheet shows that the drum must be re-examined.
4. All count sheets (for drum, can, and Helix counters) are now reviewed by a knowledgeable technical person before releasing a drum to waste.

5. All bags cut from the production line are now surveyed using gamma and neutron survey instruments. From these surveys, a plutonium value is assigned and a running inventory is kept on each drum. Any package higher than 125 mr/hr in gamma plus neutron must be placed back in the line to determine the source of the radiation. The source must be removed before the waste or residue can go into a drum.
6. First line supervision reviews drum contents and certifies total plutonium content before the drum is sealed and sent to the drum counter. Gross differences between the counter and the estimate are reviewed. Gross differences may be defined as drums shown containing zero grams by supervision, but found containing 30 or more grams by the counter.
7. Waste Management inspection procedures have been implemented which would have probably detected these two drums prior to shipping to Idaho.

# FIGURE 1

## BARREL COUNTER

BACKGROUND GAMMA	2941	SAMPLE #	T064313
OXIDE STD GAMMA	49937	BACKGROUND NEUTRON	287
FLUORIDE STD GAMMA	92825	OXIDE STD NEUTRON	19862
		FLUORIDE STD NEUTRON	472776
SAMPLE GAMMA	358134	SAMPLE NEUTRON	378573
SAMPLE ATTENUATION	100	GROSS WT IN LBS	137
TARE WT IN LBS	64	NET WT IN LBS	73
GRAMS PU	-5149.86		
UNCORRECTED GRAMS PU	-5149.86		
MS CODE	330		
I. D. #	000		

DOCUMENT\*\*\*\*\*

\* 64313 \*

\*\*\*\*\*

\*\*\*\*\*

* FROM - ACCOUNTABILITY CODE	* TO - ACCOUNTABILITY CODE	* DATE
* * W/R * MATL BAL *	* * W/R * MATL BAL *	* * *
* CTL * ALOT * AREA * FUNCT	* CTL * ALOT * AREA * FUNCT	* MO*DAY* YR*
2 * 01 * 137131 * 30	2 * 01 * 037432 * 20	*01* 19* 1 *

\*\*\*\*\*

\*\*\*\*\*

* NEW ITEM	* ELEMENT	* ITEM DESC	* NET WT	* ASSAY	* SS NET
* IDENT #	* CODE	* CODE	* K-GMS	* G/G	* WT
* 53	* 330	* 33.1	* 0.0	* 0.0	* 0.0

\*\*\*\*\*

LESS THAN AEC DISCARD LIMITS

\*\*\*\*\*

*ISSUED BY	**RECEIVED BY
*AUTHORIZED	**AUTHORIZED

\*\*\*\*\*

## KEYPUNCH

FIRST CARD-----COL. 79-80, 10  
SECOND CARD-----COL. 79-80, 20

FIGURE 2

## C BARREL COUNTER

BACKGROUND GAMMA	2941	SAMPLE #	T064315
OXIDE STD GAMMA	49937	BACKGROUND NEUTRON	287
FLUORIDE STD GAMMA	92825	OXIDE STD NEUTRON	19862
		FLUORIDE STD NEUTRON	472776
SAMPLE GAMMA	395667	SAMPLE NEUTRON	514572
SAMPLE ATTENUATION	100	GROSS WT IN LBS	150
TARE WT IN LBS	64	NET WT IN LBS	86
GRAMS PU	-2136.79		
UNCORRECTED GRAMS PU	-2136.79		
MS CODE	330		
I. D. #	000		

\*\*\*\*\*  
DOCUMENT\*\*\*\*\*

\* 64315 \*

\*\*\*\*\*

\*\*\*\*\*  
\* FROM - ACCOUNTABILITY CODE \* TO - ACCOUNTABILITY CODE \* DATE \*  
\*\*\*\*\*  
\* \* W/R \* MATL BAL \* \* \* W/R \* MATL BAL \* \* \* \* \*  
\* CTL \* ALOT \* AREA \* FUNCT \* CTL \* ALOT \* AREA \* FUNCT \*MO\*DAY\* YR\*  
\*\*\*\*\*  
2 \* 01 \* 137131 \* 30 \* 2 \* 01 \* 037432 \* 20 \*01\* 19\* 1 \*  
\*\*\*\*\*

\*\*\*\*\*  
\* NEW ITEM \* ELEMENT \* ITEM DESC \* NET WT \* ASSAY \* SS NET \*  
\* IDENT \* \* CODE \* CODE \* K-GMS \* G/G \* WT \*  
\*\*\*\*\*  
\* \* 53 \* 330 \* 39.0 \* 0.0 \* 0.0 \*  
\*\*\*\*\*

LESS THAN AEC DISCARD LIMITS

\*\*\*\*\*  
\*ISSUED BY \*RECEIVED BY \*  
\*AUTHORIZED \*AUTHORIZED \*  
\*\*\*\*\*

## C KEYPUNCH

FIRST CARD-----COL. 79-80, 10  
SECOND CARD-----COL. 79-80, 20

PLUTONIUM MEASUREMENT IN IDAHO

WASTE RETURN

R. E. Giebel  
R. G. Leebl  
J. A. Battaglino.

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Rocky Flats Division  
Golden, Colorado

Distribution:

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## PLUTONIUM MEASUREMENT IN IDAHO

### WASTE RETURN

#### INTRODUCTION

Rocky Flats routinely transfers plutonium waste to Idaho for interim storage. The barreled waste is randomly spot-checked for plutonium content by Gulf-Atomics using radiometric counting technique. Several barrels were observed to contain plutonium above the discard limits. The contents from these drums were placed into four pressure cookers by Rocky Flats personnel for return to the Rocky Flats plant. The pressure cookers were transported in DOT approved 20/30 shipping containers. The process waste material was radiometrically gamma-neutron counted upon return to Rocky Flats. To arrive at an accurate plutonium value for each pressure cooker, the contents were converted to an ash. The analyzed ash was returned to Chemical Operations for recovery. This report describes the processing of the waste material to a form applicable for plutonium measurement and summarizes the plutonium values obtained.

#### RESULTS

A detailed description of the processing scheme followed for each pressure cooker is presented in the Appendix. The contents of pressure cooker 7959B were subdivided into

five categories for processing because of its high plutonium content. Each category was gamma-neutron counted prior to being ashed for x-ray analysis. After being sampled, the ash from the five waste categories was combined and was analyzed as a single batch. The plutonium values obtained are listed in Table I.

Table I

Plutonium Results from Pressure Cooker 7959B

<u>Waste Category</u>	<u>Gamma-neutron Pu Determinations (g)</u>	<u>X-Ray Pu Determinations (g)</u>
Paper and Plastic	220 (-1-)	320
Fulflo Filter Element	47	35
R-6 Filter Pads	244 (-1-)	207
Bottle with Sludge (1st)	104	221
Bottle with Sludge (2nd)	<u>100</u>	<u>191</u>
Total (s)	715	974 <sup>(919)</sup> (-2-)

(-1-) Estimated plutonium values. Can counter is not capable of assigning a value over 200 gms.

(-2-) Composite plutonium value.

The contents of each remaining pressure cooker were measured prior to ashing by radiometric (gamma-neutron) can counting. These radiometric plutonium values, and the plutonium values obtained by x-ray analysis on the ashed materials are compared in Table II.

Table II

Comparison of Plutonium Results				DRUM COUNTER	
<u>Cooker Number</u>	<u>Can Counter (g)</u>	<u>ARCO</u>		<u>OLD</u>	<u>NEW</u>
		<u>X-ray (g)</u>			
7959A	262	267	264	166	157
7959B	715	800	974	2200	628
7961A	243	234	252	267	225
7961B	<u>106</u>	103	<u>99</u>	86	61
Total (s)	1326		1589		

#### APPENDIX

##### General Process Procedure

Each of the four pressure cookers (Type DPV-B) was shipped to Dow Rocky Flats in Type 20/30 shipping containers.

Each pressure cooker, containing process waste, was gamma-neutron counted at Idaho by Gulf Atomics. After receipt at Dow Rocky Flats, each pressure cooker was gamma-neutron counted within the 20/30 shipping container using both the old and new drum counters. Then each cooker was placed in a 55-gallon drum and again counted using the old and new drum counters.

Each cooker was then opened and the contents were examined. One cooker showed pressure on the gage and the entire cooker was placed in a glovebox to minimize accidental spread of plutonium contamination. The gage on another cooker indicated pressure but no pressure was released

when the relief valve was vented. However, when the cooker seal was broken, a gas was released and the gage returned to zero. The relief valve was defective. A third pressure cooker gage showed several pounds of pressure. The gases were released to the area ventilation system after sampling showed no radioactivity. The contents of each cooker were photographed, repackaged into one-gallon containers and gamma-neutron counted for plutonium determinations. The one-gallon containers were placed in a glovebox and separately calcined to an ash which was blended, weighed, and sampled for plutonium and americium.

#### Cooker 7959A

Cooker 7959A contained a one-gallon tin can (paint-type) which contained a polyvinyl chloride (PVC) cut-out bag. (See Figure 1, left side). The bag was placed in a glovebox and opened. It contained sludge and rags in a one-quart plastic container (See Figure 2). The contents were can-counted, calcined, weighed, and sampled (See Table II for results).

#### Cooker 7959B

The pressure gage on this cooker indicated one pound pressure. The entire cooker was placed in a

glovebox and opened. A sealed plastic outer bag and an inner bag were not pressurized; however, the inner bag was wet with droplets of a liquid.

Figure 3 shows these cut-out bags after opening.

The contents of the inner bag, as shown in Figure 4, contained three R-6 filter pads, one Fulflo<sup>®</sup> filter cartridge, a 500-ml poly bottle one-half full of wet sludge, a 500-ml poly bottle one-quarter full of sludge, loose wet sludge, plastic, and paper. A material identification ticket indicated the wet material was packaged on January 18, 1971 from

Line 2. A total of 70 ml of acid solution ( $2.2 \text{ N H}^+$ ) was collected from the plastic bags. Laboratory analyses indicated 0.21 g/l Pu and  $5.3 \times 10^{-3}$  g/l americium-241. The contents were can-counted in five packages as follows:

1. Plastic, paper, wet sludge
2. Fulflo filter cartridge
3. R-6 polypropylene filter pads
4. 500-ml poly bottle with sludge (1st)
5. 500-ml poly bottle with sludge (2nd)

Each of the five packages was individually calcined and the resulting ash was sampled. The ash was combined, blended, and a composite sample was taken for plutonium assay. Table I summarizes the plutonium values determined for each package within cooker 7959B.

Cooker 7961A

The pressure gage on this cooker indicated greater than 15 psig. The relief valve did not function. The cooker contained a large plastic bag (Figure 1, right side) which was placed in a glovebox and opened. The attached material control ticket contained no information. Two smaller plastic bags were within the large bag. One bag contained a dirty towel, the other contained some scrap metal, paper, sludge, and a large plastic tubing (Figure 5). The two packages were can-counted separately. The metal was leached and the solution was combined with the sludge, calcined to dryness, weighed, and sampled. (See Table II for plutonium results).

Cooker 7961B

The pressure gage on this cooker was under 7 psig. The cooker contained a plastic bag which appeared pressurized and was immediately placed in a glovebox. The inner plastic bag contained filter sludge (See Figure 6). The contents were calcined, weighed, and sampled (See Table II for plutonium results).